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SURFACE STUDIES

H. Fritzsche

Chicago University

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SURFACE STUDIES

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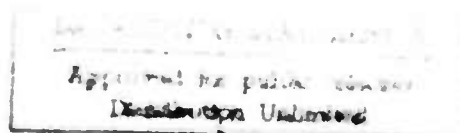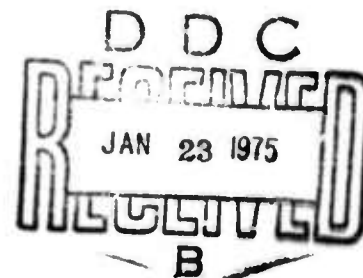Principal Investigator: H. Fritzsche - (312) 753-8221

The University of Chicago

The Division of the Physical Sciences

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I. REPORT SUMMARY

The objective of the Surface Studies Program is (i) to elucidate the inter-actions causing atoms and molecules to bind to metal surfaces and (ii) to study the dynamics of gas-solid interactions on an atomic scale, and with an accuracy which has not been possible heretofore. This is required for developing a theoretical understanding of the fundamental processes underlying catalysis, surface electro-chemistry, adhesion, friction, corrosion and the operation of fuel cells.

These objectives are being pursued in a collaborative effort by using several different research tools. The correlation of the observations obtained by these different techniques is an important element of this cooperative study. A significant theoretical effort centered on the electronic structure of transition metal surfaces and on chemi-sorption supports our experimental work.

The basic parts of our Surface Studies Program are in brief the following.

A. Adsorption Studies.

Modern research in chemisorption has revealed the existence of a multitude of different binding states on single crystal planes in many chalcogenide systems. Informa-tion on the number density, existence range, and interconversion of these states is beginning to accumulate but the detailed electronic and geometric structure of the adsorption complexes in various binding states is largely unknown. The main purpose of the effort described here is to elucidate the electronic, and through that, the geo-metric structure of chemisorption complexes. In order to achieve this, apparatus is needed in which a number of different experiments can be carried out in reasonably rapid succession on the same substrate under constant conditions of adlayer history.

We have designed and in large part completed a stainless steel ultrahigh vacuum system which enables us to carry out in the same apparatus on a given sample the following measurements: low energy electron diffraction, ultraviolet and X-ray photoemission spectroscopy, Auger analysis, electron impact desorption, work function measurements, and inelastic electron reflection spectroscopy. The system consists of a modified Varian Leed system, a Physical Electronics, Inc. double pass cylindrical mirror analyzer and X-ray source, and a Vacuum Generator Ltd. vacuum ultraviolet lamp. A quadrupole mass analyzer serves for residual gas analysis as well as for electron impact desorption measurements. A sample holder capable of allowing the samples to be cooled to near 20 K or heated to 2500 K has been designed and is in the process of construction. Further construction details of this system are discussed in Section II.1 of this report.

B. Field Emission Spectroscopy.

The energy distribution of field emitted electrons for the 110 plane of W covered with Au has been obtained. [1] There is an increase in the work function and a general enhancement in emission; the distribution is otherwise structureless, indicating a very broad s-like resonance for Au adsorbed on this plane of tungsten.

By analyzing the time correlation of current fluctuations in field emission we have been able to determine the surface diffusion mobility of adsorbates. [2] Apparatus for carrying out these measurements has been constructed and debugged and measurements for CO on tungsten are currently in progress. Data obtained so far agree well with deductions about CO binding states made from other measurements. [3-5]

We have started experiments to demonstrate the feasibility to extend the range of

conventional field emission spectroscopy from the limited energy range of 2-3 eV below the Fermi level of the field emitting surface to beyond 5 eV below the Fermi level. This new technique of extended range field emission is possible by using a system which accelerates rather than decelerates the field emitted electrons before energy analysis. The advantage of an accelerating technique is that only one beam cross-over occurs in the electron optics, and that in space charge effects are negligible in that cross-over region because of the high energy (10-25 keV) of the electrons. See Section II.2 for more details.

C. Direct Electron Micrographic Observation of Adsorption and Diffusion.

An important novel aspect of the use of the scanning electron microscope of high resolution, designed by A. V. Crewe et al. at this University, [6,7] is the possibility of studying diffusion and nucleation of clusters of heavy atoms on substrates made of lighter elements. Since one can actually see individual heavy atoms on these thin substrates, one can measure atom-to-atom distances and thus directly obtain binding site information. We have observed atom "hopping" from site to site within the time of successive exposures. These hopping distances can be determined for intervals as small as 3 $\overset{\circ}{A}$. One can thus determine local directional effects and short-range order effects upon diffusion. Substrate order is determined in situ by obtaining scanning electron diffraction patterns of particular areas of interest. The selected area can be as small as the diffraction conditions permit. See Section II.3 and Figs. 2 and 3 for preliminary results of this new technique.

D. Dynamics of Atom and Molecule Collisions with Surfaces.

The motivation for these studies is the need for reliable information on the processes of absorption, adsorption, reaction, and reflection af (gas) molecules when they strike a surface. Such information can contribute to understanding and discovery in the fields of catalysis, energy conversion, corrosion, heat transfer, aerodynamic drag, and surface reactions. Until quite recently it was difficult ta conceive of a feasible apparatus that could provide the flexibility in the ultrahigh vacuum region to measure the dynamic parameters of velocity, angle, and rate, and to detect chemical species. Advances in high resolution gas phase molecular beam scattering, in achieving the cleanliness required to prepare and maintain a surface, and some pioneering studies in this field are guiding us in the present construction of a universal solid-gas molecular beam apparatus.

The most important single feature af this apparatus is a universal molecular detector of high selectivity and sensitivity that can detect whatever process is to be observed without requiring long times of observation or changes in surface coverage. For this a high sensitivity mass spectrometer detector will be placed in an ultra high vacuum system that is compatible with these ends, and which permits movement af the detector in close proximity to the surface being observed.

Specifically the machine will have two molecular beam sources for dosing a solid sample surface, velocity selectors for the incoming and desorbing beams, sample temperature controls, sample and detector motions that provide for wide examination of angular dependent features, and a surface Auger analyzer, all enclosed in a bake-able ultrahigh vacuum system. The design provides as intense and clean signals of the

scattered beams as is possible by a relatively small 10 cm spacing between solid sample and electron bombardment region of the quadrupole mass analyzing detector. There is a time-of-flight velocity selector and a doubly differentially pumped detector manifold in this 10 cm space. Provisions are made for supersonic and effusive velocity selected sources at distances 25 cm from the sample surface. These sources will be differentially pumped to eliminate diffusely directed gas.

The layout for the molecular beam surface machine is completed. Drawings of the main tank have been released for fabrication. Bids for pumps have been received. Rotary seals have been designed to permit rotation of 10" flanges. Internal layout to permit the smallest possible beam paths, consistent with the required functions and simplicity has been evolved. The following subsystems are in active design: bakeout oven, high speed chopper, detector, and crystal mount. See Section II.4 and Fig. 4 for details.

E. Physical and Chemical Processes near Metal Surfaces in High Electron Fields.

Some time ago M. G. Inghram discovered [8-10] the existence of fine structure in the energy distribution of ions produced by field ionization. He suggested that this structure is due to field induced resonance states at the surface of the electrode, the "Müller tip", which produces that field. Recently he discovered that part of the structure in the energy distribution of the ions produced in the high field near the tip of the electrode is due to field dissociation of excited ions. Specifically in dissociative field ionization of HD at the critical distance from the surface, the H^+ ions exhibit structure in the energy distribution which does not exist in H^+ from H_2. He suggested that this structure results from an interaction of the vibration of the molecule

with field induced rotation. [11] This possibility has been further studied both experi-mentally and theoretically. [12]

F. Theoretical Studies.

Problems of current interest in the theoretical group include many-body and nonorthogonality effects in chemisorption, particularly the effect of interactions between electrons in the substrate, models for molecular chemisorption, and the col-lective dielectric and magnetic properties of both clean and adsorbate-covered surfaces. S. K. Lyo and R. Gomer have attempted to make a quantitatively meaningful descrip-tion of H chemisorption on the 100 face of tungsten, extending the Newns Hartree-Fock theory to include the continuum wavefunctions in the basis along with the metal d states, and taking account of the nonorthogonality of the adatom orbital to these states. Screening in the metal was treated phenomenologically using a classical image potential, which has the effect of reducing the effective interaction strength on the adatom. The description provided a reasonable explanation of existing experimental data for this system. [13]

J. Hertz has derived equations for the electron Green's functions, both on the adatom and in the substrate, for a localized-spin-fluctuation description of the dynam-ics of chemisorption. Interactions both on and off the adatom are taken into account. This sort of approach, which has been quite successful in describing dilute magnetic alloys, is most appropriate when corrections to Hartree-Fock theory are small, but it can also be used (in a model sense) in strongly correlated systems. It allows one to see in a straightforward way how the spectrum of the adatom is modified by correlation. Illustrative calculations are in progress, in collaboration with J. Handler.

Another approach to chemisorption in the presence of strong correlations is
afforded by a generalization of the so-called Hubbard decoupling procedures for the
equations of motion for the Green's functions, as done by Hewson for the magnetic
impurity problem. J. Bell and J. Hertz have constructed the electron Green's func-
tions within this approximation for the chemisorption problem, once again including
interactions both on the adatom and in the substrate.

Theoretical and experimental aspects of chemisorption have been reviewed by
R. Gomer. [14,15]

II. TECHNICAL PROGRESS

1. The Leed-Auger-UPS-Esca-EID Machine.

This apparatus consists of a stainless steel ultrahigh vacuum system which will
permit different measurements to be carried out under identical conditions on the same
substrate. The measurements available will consist of low energy electron diffraction,
Auger analysis, inelastic electron reflection spectroscopy, ultraviolet and X-ray
photoemission spectroscopy, and electron impact desorption. Fig. 1 shows schematic-
ally the layout of the various detector and electron-X-ray and ultraviolet sources.
Connections to the sample are made via flexible Cu leads which permit 360° rotation
of the sample and cooling by means of a small liquid H_2 or N_2 reservoir, located
inside the vacuum system.

To date the basic vacuum system has been obtained from Varian, together with
the Leed optics and associated electronics. The Leed optics will also be used for ultra-
violet photoemission spectroscopy. The light source, obtained from Vacuum Generators,
Ltd. has now been received. A quadrupole mass analyzer which will be used for residual
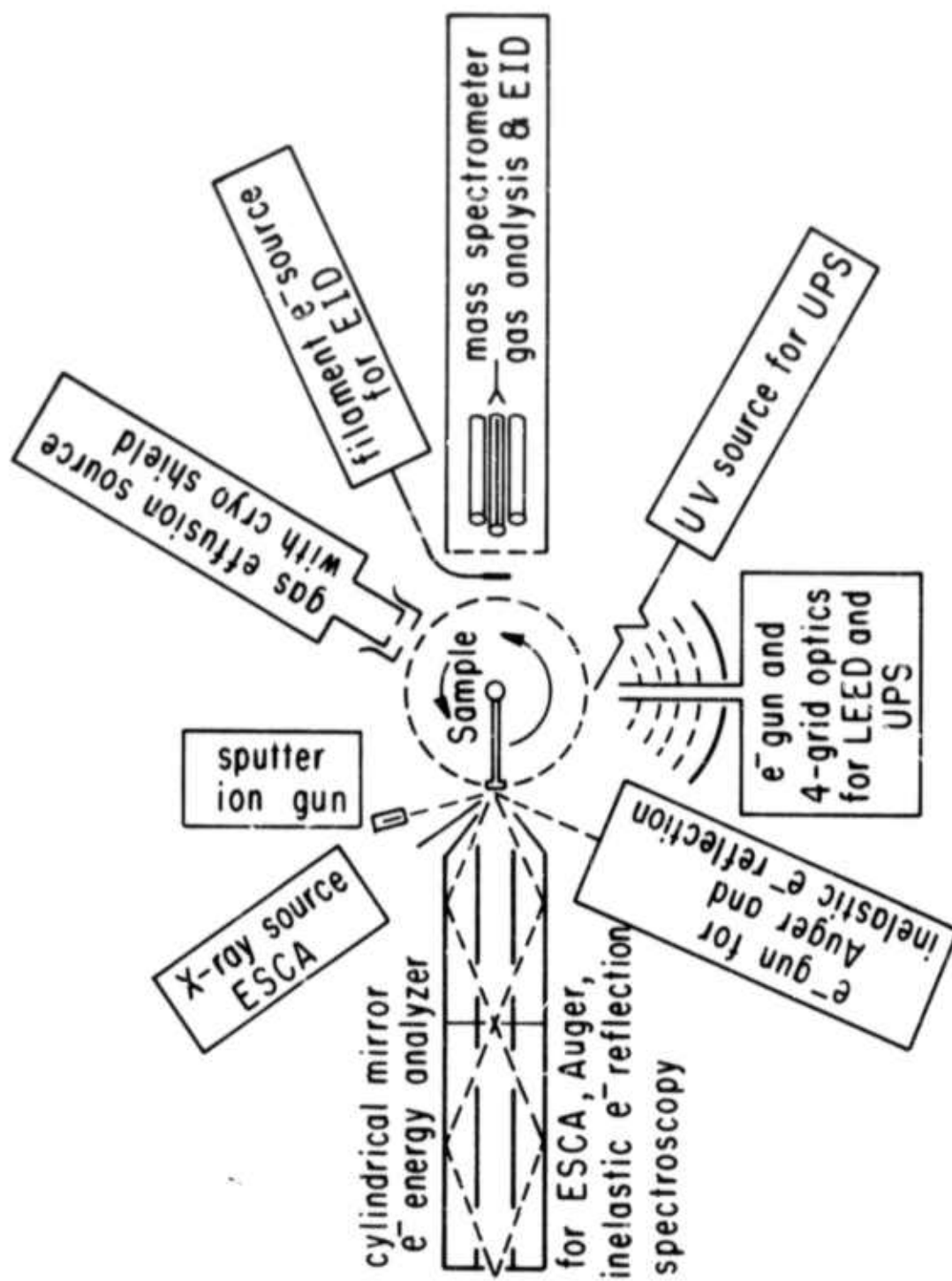
Fig. 1 Schematic Layout of LEED-Auger-UPS-ESCA-EID Machine

gas analysis as well as for electron impact desorption measurements has been obtained from UTI, Inc. A double pass cylindrical mirror electron energy analyzer from Physical Electronics, Inc. has also been received. The X-ray source which will be used for X-ray photoemission spectroscopy in conjunction with the mirror analyzer has not as yet been received from Physical Electronics, and constitutes the last major item of equipment for which we are waiting.

Currently the gas inlet and differential pumping system for the windowless, differentially pumped ultraviolet light source has been designed and is under construction. A sample holder which will permit cooling the sample to 20 K or heating it to arbitrarily high temperatures has been designed and is being constructed.

A gas effusion source has been designed and constructed. An add-on modification, which will convert the gas inlet into a true molecular beam source is in the design stage and will be constructed shortly. This modification consists of a liquid H_2 cooled cylinder with an exit slit located in front of the effusion source. The function of this cooled section is to intercept all effusing gas except that directly headed for the sample surface. In this way the latter can receive a deposit without introducing gas into the main vacuum system, thereby making it possible to obtain partial coverages at will; this method also avoids the possibility of gas exchange on the walls of the vacuum system, since the pressure in the effusion source is so high that effects of this kind are negligible.

When this equipment is completed and tested it is planned to investigate first the electronic structure of CO binding states on tungsten by means of UPS and ESCA experiments. Although considerable work of this kind has been done by various groups during the last year, many of the experiments have been performed on layers deposited at room

temperature, or during cooling after flashing the substrate in ambient CO. Since previous work in our and other laboratories indicates the simultaneous existence of several binding states under these conditions, as well as coverage-sensitive features in the electronic structure, it is considered worthwhile to carry out further experiments with emphasis on the cleanest possible separation of states. If possible we hope also to work at various coverages. The gas inlet system is being especially designed to make this possible.

It is also planned to investigate in this way ginding modes created by electron impace, i.e. not producible thermally.

After completion of this work it is planned to carry out similar studies for nitrogen adsorption on tungsten. Finally an attempt to investigate chemical shifts in alkali and alkaline earth adsorption be means of ESCA and possibly by inelastic electron spectroscopy will be made. The point of this work is to obtain absolute values of adsorbate charge and information on screening lengths at metal surfaces.

2. Extended Range Field Emission Spectroscopy.

Field emission spectroscopy (FES), i.e. the analysis of energy distribution of field emitted electrons, is one of the most direct means of determining the local density of states at an adsorbate, and consequently of great potential usefulness for elucidating the electronic structure of adsorption complexes. Its principal drawback to date has been the limited energy range (abc 2-3 eV below the Fermi level) which can be probed. The reason for this is that the field emitted current is a steeply decreasing exponential function of the energy below the Fermi level so that if only 0.1% of the electrons at the Fermi level get inelastically scattered somewhere in the analyzer system and lose a few

eV of energy, they overwhelm the true signal at the detector for $E \gtrsim$ 2–3 eV, E being the energy below the Fermi level.

All energy analyzers used for FES up to the present have operated by decelerating field emitted electrons passing through a probe hole from several keV to at most 1–2 eV, in order to achieve good resolution with analyzers having $\Delta E/E \sim 0.03$–0.1. This technique, however, aggravates several of the principal causes of the spurious electrons appearing at the analyzer detector: (i) multiple beam cross-overs occur with low voltage electrons where electron-electron scattering, i.e. space charge becomes troublesome; (ii) multiple aperture systems are necessary for the defining optics and result in excessive electron scattering from the apertures; and (iii) the optical properties of these decelerating systems are complicated functions of the field emission voltage.

We plan to increase the energy range (to $E \gtrsim$ 5 eV) and therefore the usefulness of FES by utilizing a system which accelerates rather than decelerates the field emitted electrons before energy analysis. The advantage of an accelerating technique is that only one beam cross-over need occur, and that the electrons will have high energy ($\sim 10 - 25$ keV) in the cross-over region so that space charge effects are negligible. Moreover, this cross-over can be as small as 100 Å in diameter by using an optimally designed acceleration system.[16] It then serves as a real point source for an energy analyzer and thus eliminates aberrations due to finite source size. In such a system, the only defining aperture is at the exit of the acceleration system, and electrons scattered at this aperture can effectively be removed by placing a much smaller aperture at the beam cross-over.

The first order optics of such a system depends only upon the ratio of the

accelerating voltage to the field emission voltage. [16] This method, of course, requires an electron spectrometer of close to 1 PPM energy resolution. However this capability has already been demonstrated at The University of Chicago. [17]

We have initiated a program to demonstrate the feasibility of extended range FES using an accelerating system by utilizing one of the scanning electron microscopes in the laboratory of A. V. Crewe. However, the instrument which we are currently using was not designed for FES and is suitable at best for demonstrating the feasibility of the method. For instance, it is set up for only 50-75 meV resolution; the field emitter can only operate at room temperature; the system is not bakeable and the vacuum near the tip is only $\sim 3 \times 10^{-9}$ torr; there is no deflection system around the field emitter to permit energy distribution to be obtained from several crystal planes under the same conditions; and it is not easy to deposit adsorbate gases onto the field emitter.

We have obtained some preliminary results from this system at present and expect to obtain a concrete demonstration of the feasibility of extended range FES in the early part of 1975. We plan to construct (in the 1975-76 period) a dedicated extended range FES spectrometer based on the accelerating system described above and specifically optimized for FES of adsorption complexes. The instrument will be a bakeable stainless steel system to allow flexibility and will be designed for 20 meV resolution. The emitter will be coolable to liquid He or H temperatures and the system will be capable of operating with high field emission currents (i.e., high electric fields) thus decreasing the exponential slope of the field emission distribution below the Fermi level and further increasing the energy range below the Fermi level which can be probed.

3. Direct Electron Micrographic Observation of Adsorption and Diffusion.

The first direct observation of adsorption and diffusion of heavy atoms on thin evaporated substrates was achieved by M. Isaacson and A. V. Crewe. [6] They used a transmission electron microscope [7] with an electron beam probe diameter at the specimen of 2.5 $\overset{\circ}{A}$. Their preliminary results indicate that the observed atom motion is due to surface diffusion and not induced by the electron beam. So far, all binding density and motion analyses have been done relatively crudely by hand measurements of micrographs (see Figs. 2 and 3). In the near future we plan to extend the temperature range for diffusion and to analyze the micrographs with automated x-y scanning tables commonly used for cloud or spark chamber photographs. Having the atom coordinates on tape allows computer analysis of interatomic separations, hopping velocities, and probability of cluster formation.

This new method for studying chemisorption complements our other techniques because of the following factors. First, the thin evaporated substrates initially consist of B, C, Al_2O_5 and Si; materials which are not easily fabricated into field emitters and are therefore not readily accessible for study using field emission techniques. Second, the evaporated substrates can be either homogeneous or heterogeneous and can be made as either amorphous or crystalline films. The requirements for an atomically smooth single crystal surface is less severe in the STEM than for photoemission (or reflection) studies of adsorption, since crystals as small as several hundred angstroms in size will suffice for our studies.
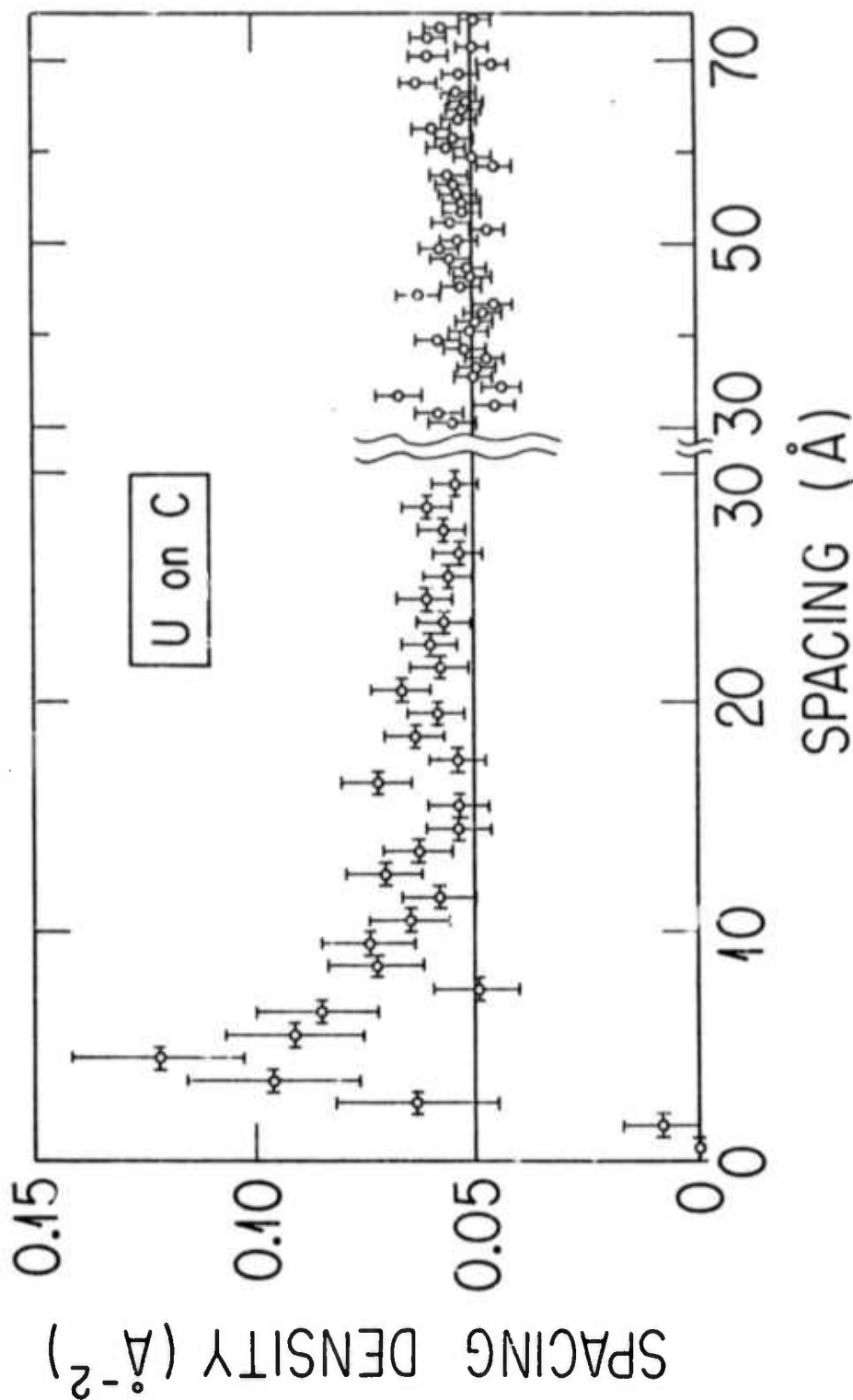
Fig. 2. Spacing density for uranium atoms on amorphous carbon substrates (20 Å thick). The plots represent the number of atoms per Å2 at a given distance from each atom. The number of atoms analyzed was 911. The error bars represent one standard deviation. The solid line is the expected distribution for randomly arranged atoms.
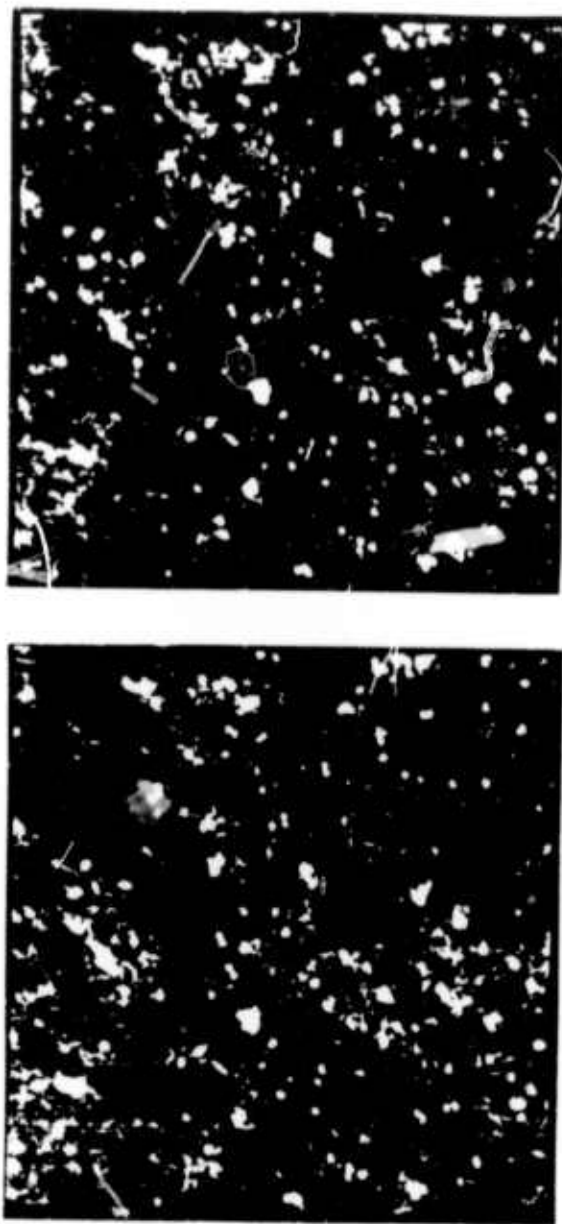
Fig. 3. Example of a field of view of uranium atoms on a thin carbon substrate which was analyzed to obtain the spacing densities above and frequency of motion. The micrographs are 195 $\overset{\circ}{A}$ full scale and the time between micrographs was 25 seconds. The micrographs were obtained using electrons scattered elastically from the specimen.

4. Universal Solid-Gas Molecular Beam Apparatus.

Presently our understanding of gas phase chemical reactions and physical processes is being revolutionized by crossed molecular beam experiments. These are clarifying the nature of molecular collisions by preparing reacting gas molecules in precisely known initial states, permitting them to collide, and observing the consequences of collision by the identification of the final states.

We intend to employ these powerful and precise techniques to the study of surface gas reactions. For this purpose we are constructing a molecular beam machine using a surface as the target. In order to adapt the beams technique to the study of surfaces one must work in a vacuum environment that is typically three orders of magnitude better than used in gas-gas collision studies. This will preserve surface cleanliness during the experiments. The present research then addresses itself to the design of an apparatus that is capable of reaching 10^{-10} torr., and yet which has the features which provide the flexibility and analyzing capability of present molecular beams apparatus. These include beam velocity analyzing capability, high sensitivity universal detectors that are capable of covering the widest range of scattering angles, plug-in sources, and minimal beam travel distances to provide maximum signal intensity. Such an apparatus must have additional features which address themselves to surface orienting, surface cleaning, surface analysis, and surface temperature control.

The apparatus under construction is illustrated in Fig. 4 which shows the location of the main instrumental features. It comprises two fixed plug-in sources on a horizontal plane, with room for velocity selection of these beams, a rotatable differentially pumped electron bombardment quadrupole mass analyzing universal detector, velocity analysis of
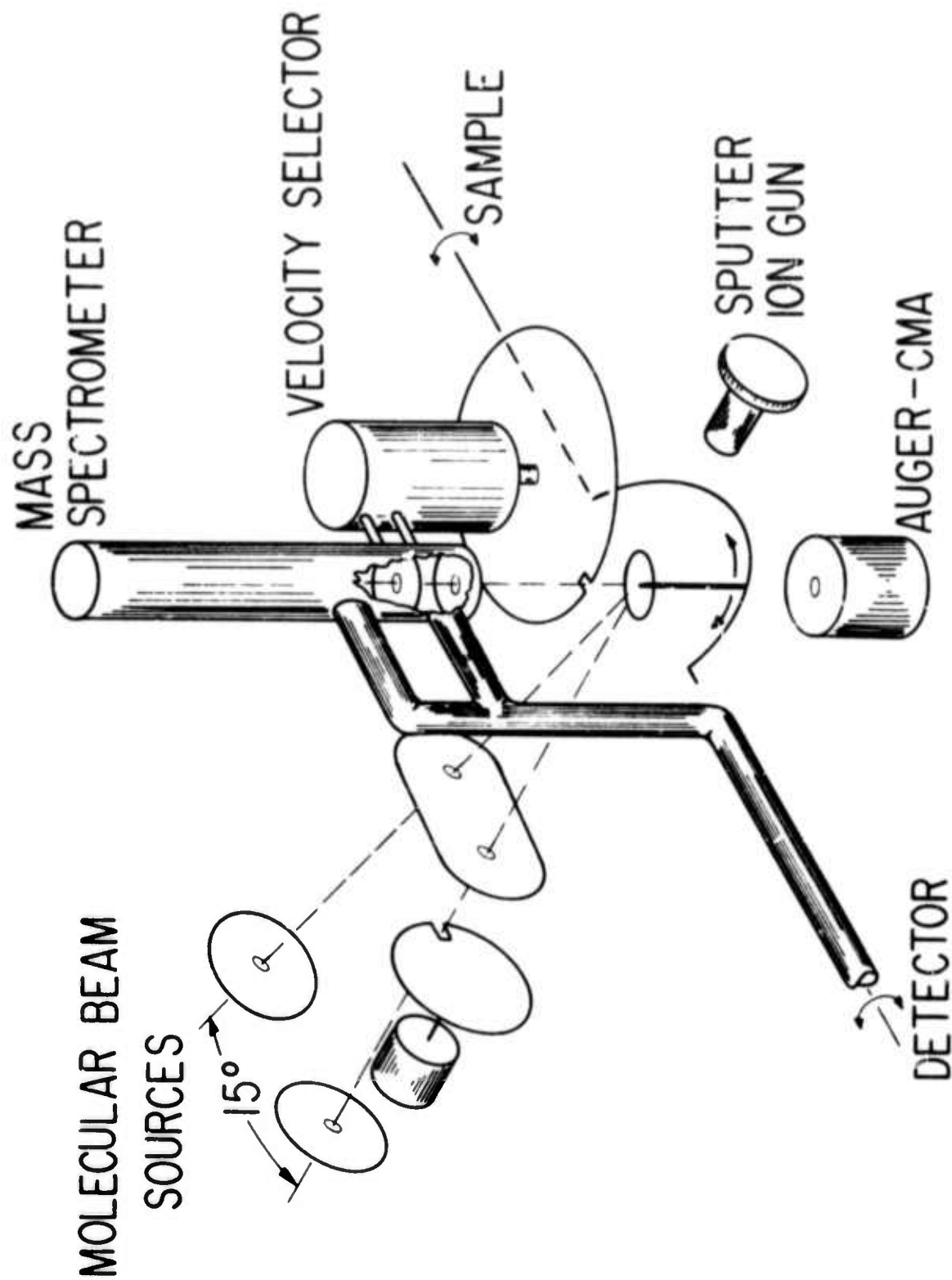
MOLECULAR BEAM
SOURCES

15°

MASS
SPECTROMETER

VELOCITY SELECTOR

SAMPLE

SPUTTER
ION GUN

AUGER—CMA

DETECTOR

Fig. 4 Schematic Layout of Surface – Molecular Beam Appacatus

the detected beam, a rotatable sample holder, sputter cleaning gun, ond an Auger electron CMA surface analyzer. A horizontal plane of the axis of rotation of sample and detector was chosen to make possible studies of liquid samples with an auxiliary vertical beam source. The minimal sample-detector distance of 10 cm was chosen to be smaller than the minimal source-sample distance of 25 cm because of the overriding necessity for sensitivity of detection of desorbed species. It should be possible to detect 10^{-7} to 10^{-5} monolayers desorbing per second, or in other circumstances 10^{-7} to 10^{-5} product conversion of an incoming reactant.

High Sensitivity Detector. We have chosen to use a quadrupole electron-bombardment-ionizing mass spectrometer. High sensitivity will be achieved primarily by placing the ionizing region of the detector very close to the solid sample, 10 cm in this instance. This will provide a very high sensitivity against $1/R^2$ losses and against small changes in surface coverage. Another factor in achieving a high sensitivity will be a high rejection of background molecules coming from directions other than from the solid sample. This is to be achieved by two stages of differential pumping with shaped interchamber slits. The detector pumps will be mounted externally to the chamber through the exploitation of a large UHV rotary seal. The detector will be rototable about the solid sample in a plane and this 140° sweep of the 60 cm long detector determines the major dimensions of the experimental molecular beam tank.

Plug-In Sources. Because the detected signal intensity for a single source experiment will depend on the source-to-sample distance to the 2nd power as contrasted to the 4th power for beam-beam scattering experiments, it was felt that the sources could be placed at a longer distance (25 cm) than the detector. Also supersonic

beam sources can provide intensities upwards of one monolayer per second at these distances which is believed to be sufficient for most experiments anticipated. These were the considerations in the necessary trade-off in the sample-to-source versus sample-to-detector spacing. One of the sources will shoot in the plane of rotation of the detector and the other will shoot at an angle of 15 degrees from this plane. It is intended to build up and add to our present technology of effusive, supersonic, and seeded supersonic beams. One will want to cover as wide a source temperature range as possible and to be capable of providing electrical discharge sources where needed.

Vacuum System. To achieve a target pressure of 10^{-10} torr. we have selected differential diode ion pumps. The material of construction of the molecular beam chamber, 304 stainless steel, will be vacuum outgassed at a temperature of about $700^{\circ}C$ for 20 hours. This has been found to be effective in permanently removing dissolved hydrogen which would otherwise limit the ultimate vacuum. For each cycle of pump-down from atmosphere the system will be baked at up to $250^{\circ}C$. This bake-out will proceed with air inside the chamber for the first few hours before vacuum is drawn. These heat processing techniques have been shown by the research group at CERN to give excellent vacuum performance. The parameters of our system will enable a 24 to 48 hour pump-down from atmosphere to operating pressures. The chamber will be roughly rectangular 4' x 3' x 2' and will weigh roughly 3500 lbs. The detector chambers within this molecular beam chamber will be isolated by a vacuum tight valve and pumped separately to keep the background pressure low.

Surface Analysis and Cleaning.   We plan to use a commercially available single cylindrical mirror (CMA) Auger analyzer for detecting atomic impurities on solid sample surfaces. A 1 to 3 kV sputter cleaning gun will be mounted so as to be used with the CMA. A means for bleeding in oxygen and other surface treating gases will be provided. The crystal mount will have provision for high temperature flash cleaning of the solid samples.

Sample Holder. The sample holder will provide the possibility for plug-in installation of premounted crystals.   The mounts will provide for temperature variation from liquid hydrogen temperatures to the melting of sublimiation temperatures of the solid sample. A pantograph mechanism will provide for out-of-plane tilting of the crystal, in addition to rotation coaxial to the detector axis of rotation that a rotary UHV seal provides. The pantograph provides clearance that is required for the rotation of the detector and the detector velocity selector.

Velocity Selectors. In addition to providing rapid enough chopping rates to analyze the velocity of the moving atoms or molecules provision must be made to spin disks approximately 6 in. in diameter at speeds of 20,000 RPM. This will be achieved with a drive unit that is independently pumped with two stages of differential isolation. This unit is designed for water cooling of the bearings when the molecular beam chamber is undergoing its 250°C bake-out cycle.

Present Status. Machining of the main chamber has begun. Gaskets and bearings for rotary seals are ordered. Detector design details are 60% completed. Sample mount details are 30% completed. Bake-out procedures and oven design are about 50% completed. Auger and sputter gun are on order.

Experimental Program. Experiments on the variation of sticking coefficient with degree of surface coverage will be performed first. These experiments will detect the intensity of molecules scattered from the surface as the surface is exposed to an increasing dose from a molecular beam. By varying the detector and crystal orientations it will be possible to observe the angular distribution of the scattered molecules.

Velocity and energy accommodation experiments will entail velocity selected dosing beams and velocity analysis of the scattered molecules. The velocity and internal temperature of the incoming molecular beam will be varied as well as the temperature and orientation of the solid target. A chemical reaction such as a surface oxidation can be observed with one gas phase reactant. More complicated systems involving two gas phase reactants can be studied using two molecular beams simultaneously. The influence of surface diffusion on such reactions may be explored by separating the regions of the solid sample which the two different beams strike. Velocity analysis of products of such reactions will measure the degree of equilibration of product molecules with the surface.

Diffraction and selective trapping of noble gases will prove useful as a diagnostic for surface condition. Velocity analysis of the scattered particles help to distinguish both elastic and inelastic processes and thereby increase our knowledge of details of surface structure and the modes by which energy is transferred and stored in the solid.

It should be possible to explore by techniques that are complementary to existing ones the processes of surface diffusion and thermal desorption. The correlation between residence time and the state of a desorbed particle will shed light on the dynamics of

desorption. The effects of molecular orientation upon trapping and surface reactions may also be explored with the use of inhomogeneous state selecting fields. At a later stage the effects of internal energy may possibly be explored through photoexcitation of incoming beams on surfaces. It is also contemplated that internal excitation could be accomplished by electron bombardment.

PERSONNEL

A. <u>Faculty Members</u>

Morrei H. Cohen, Louis Block Professor, The James Franck Institute, Department of Physics, and Department of Biophysics and Theoretical Biology

Robert Gomer, Professor, The James Franck Institute, and Department of Chemistry

John A. Hertz, Assistant Professor, The James Franck Institute, and Department of Physics

Mark G. Inghram, Samuel K. Allison Distinguished Service Professor, Department of Physics

Michael Isaacson, Assistant Professor, Enrico Fermi Institute, and Department of Physics

Lennard Wharton, Associate Professor, The James Franck Institute, and Department of Chemistry

B. <u>Research Associates</u>

D. Auerbach*	B. Bell
G. Hanson*	A. Madhukar
C. H. Huang*	A. Krauss*

C. <u>Graduate Student Assistants</u>

J. Chen	J. Handler*
L. Richter	G. E. Humes
C. Leung	T. Noguchi*
C. Wang	N. W. Parker
C. Becker*	M. Utlant

D. <u>Student Technicians - Part Time</u>

J. Blazey	G. Hofer*

E. <u>Instrument Designer</u>

S. Zelinski, Argonne National Laboratory

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